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A Technique for Determining the Poisson's Ratio of Thin Films

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The theory and experimental approach for a new technique used to determine the Poisson's ratio of thin films are presented. The method involves taking the ratio of curvatures of cantilever beams and plates micromachined out of the film of interest. Curvature is induced by a through-thickness variation in residual stress, or by depositing a thin film under residual stress onto the beams and plates. This approach is made practical by the fact that the two curvatures are the only required experimental parameters, and small calibration errors cancel when the ratio is taken. To confirm the accuracy of the technique, it was tested on a 2.5 μm thick film of single crystal silicon. Micromachined beams 1 mm long by 100 μm wide and plates 700 μm by 700 μm were coated with 35 nm of gold and the curvatures were measured with a scanning optical profilometer. For the orientation tested ([1 0 0] film normal, [0 1 1] beam axis, [0 $\bar{1}$ 1] contraction direction) silicon's Poisson's ratio is 0.064, and the measured result was 0.066 ± 0.043. The uncertainty in this technique is due primarily to variation in the measured curvatures, and should range from ± 0.02 to 0.04 with proper measurement technique.

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INTRODUCTION

To effectively model and design micro-mechanical devices fabricated from thin films, the mechanical properties of these materials must be known. Properties of interest include the elastic stiffnesses, thermal expansion coefficient, fracture toughness, and the residual stresses associated with deposition and thermal processing steps. It is also important to be able to monitor film properties from different depositions to ensure run-to-run uniformity. Driven by a need to acquire the elastic moduli of silica and hafnia films for models simulating the opto-mechanical failure modes of thin film multilayer mirrors, a new technique for determining the Poisson's ratio of thin films was developed. A number of methods have been published for obtaining Young's modulus, E, of thin films [1], including measurement of the resonant frequency of micromachined devices, deflection of cantilever beams using a nano-indentation instrument, and pressure-deflection behavior of bulged diaphragms. The dimensionless Poisson's ratio, ν, a measure of lateral contraction per unit uni-axial extension, is more difficult to obtain experimentally because it is a second order effect. Previously published techniques for determining the Poisson's ratio of thin films include deflection behavior of pressurized diaphragms [2-4], high resolution x-ray diffraction lattice parameter measurements of epitaxial films [5], and a method applied to polyimide films in which the in-plane stress is measured while holding the film at constant length and subjecting it to a hydrostatic pressure [6].

The technique presented here utilizes residual stresses of a thin evaporated film to apply a uniform load to cantilever beams and plates micromachined out of the film of interest, causing them to curl. Alternatively, if a sufficient through-thickness gradient in residual stress exists in the film, the cantilevers will curl upon release, making it unnecessary to coat them with the evaporated film. The beam curvatures are measured with a non-contact scanning optical profilometer, and the ratio of curvatures between beams and plates is used to calculate the Poisson's ratio. An advantage of this approach is that the calculation depends only on the two curvatures and is independent of parameters such as film thickness, Young's modulus, and applied load, as long as these quantities are uniform. In this paper, the theory and method for determining Poisson's ratio are presented, and the technique is applied to a 2.5 μm thick single crystal silicon film to confirm its effectiveness.

THEORY

For a linearly elastic isotropic material, the strain in the x-direction, ε_x, is related to the state of stress by

$$\varepsilon_x = \frac{1}{E}(\sigma_x - \nu(\sigma_y - \sigma_z)) \,, \tag{1}$$

where E is Young's modulus, ν is Poisson's ratio, and σ_x, σ_y, and σ_z are the principal stresses. For a beam with its axis in the x-direction and its width aligned with the y-direction, subjected to a stress σ in the x-direction, Eq. 1 becomes, after rearranging,

$$\sigma = \varepsilon_x E \,. \tag{2}$$

If instead the material is in the form of a plate in the xy plane which is subjected to a uniform bi-axial stress such that $\sigma_x = \sigma_y = \sigma$ with $\sigma_z = 0$, then Eq. 1 reduces to

$$\sigma = \varepsilon_x\left(\frac{E}{1-\nu}\right) = \varepsilon_x E' \,, \tag{3}$$

where $E' = E/(1-\nu)$ is referred to as the bi-axial modulus. Similarly, the curvature ρ of a beam subjected to a bending moment per width of $M_y = M$ is given by

$$\frac{1}{\rho} = \frac{Mb}{EI} \,, \tag{4}$$

while the curvature ρ' of a plate subjected to moments per width of $M_x = M_y = M$ is given by [7]

$$\frac{1}{\rho'} = \frac{Mb}{E'I} \,, \tag{5}$$

where b is the width of the beam or plate and I is the moment of inertia, given by

$$I = \frac{bh^3}{12} \tag{6}$$

for a rectangular cross section with thickness h.

For a micromachined beam and plate of the same thickness subjected to the same applied moment per unit width, taking the ratio of Eqs. 4 and 5 and solving for ν gives

$$\nu = 1 - \frac{\rho}{\rho'} . \qquad (7)$$

If the uncertainty in the radius of curvature is ± σ, then the upper and lower bounds of the Poisson's ratio calculated using Eq. 7 are given by

$$\nu = 1 - \frac{\rho}{\rho'} \pm \frac{2\sigma}{\rho} . \qquad (8)$$

The condition of a uniform bending moment is satisfied by two loading cases: a variation in residual stress through the film thickness [8], or when a thin film under residual stress σ_f is deposited onto the beam and plate. When applying Eq. 7 for the latter case, the moment M per width is

$$M = \frac{1}{2}\sigma_f th , \qquad (9)$$

assuming the film thickness t is much less than the beam and plate thickness h. However, when the film's thickness is not much less than that of the beam and plate, Eq. 9 must be modified to include the bending stiffness of the thin film, and the film's elastic moduli become new unknown variables. For a thick film with residual stress σ_f and Young's modulus E_f, deposited onto a beam with Young's modulus E_b, the curvature is given by

$$\frac{1}{\rho_{thick}} = \frac{6ht(h+t)\sigma_f}{E_b h^4 + 4E_f h^3 t + 6E_f h^2 t^2 + 4E_f h t^3 + \frac{E_f^2}{E_b} t^4} , \qquad (10)$$

a result based on an analysis by Davidenkov [9]. When t<<h, Eq. 10 reduces to the Stoney rela-

tion [10]

$$\frac{1}{\rho_{thin}} = \frac{6t\sigma_f}{E_b h^2}. \tag{11}$$

For small deformations, Eqs. 10 and 11 can be applied to the curvature of a bimorph plate simply by replacing E_f and E_b by their respective biaxial moduli E_f' and E_b' [11]. Defining the variables m = t/h, n = E_f/E_b, and k = $(1-\nu_f)/(1-\nu_b)$, and taking the ratio of Eq. 10 for a beam and a plate, the Poisson's ratio for a beam coated with a thick film is given by

$$\nu_b = 1 - \frac{k^2 + 4kmn + 6km^2n + 4km^3n + m^4n^2}{k^2(1 + 4mn + 6m^2n + 4m^3n + m^4n^2)}\frac{\rho}{\rho'} = 1 - c(k, m, n)\frac{\rho}{\rho'}. \tag{12}$$

Equation 12 can be compared with Eq. 7 to determine the error involved in making the thin film assumption. Figure 1 shows a plot of c vs. m for three values of k and n = 1, illustrating the dependence on the film thickness to beam thickness ratio. The dependence of c on n, the ratio of Young's moduli, is small compared to the dependence on k. When the film and beam have the same Poisson's ratio (k = 1), Eq. 12 reduces to Eq. 7 (c = 1) regardless of the values of m and n, justifying the use of this technique for the case when curvature of the beam and plate is induced by a through-thickness variation in residual stress.

EXPERIMENTAL PROCEDURES

While the theory presented above is straightforward, proper experimental technique is essential to obtaining accurate results. From Eq. 11, the curvature depends linearly on the film stress and the film thickness, and is inversely proportional to the square of the beam thickness. These parameters are assumed to be equivalent for the beam and plate when applying Eq. 7 to determine the Poisson's ratio. Experimentally, errors associated with film thicknesses and stress uniformity can be reduced by micromachining the beams and plates on the same wafer in close proximity to each other. The thickness and stress of the loading film should be chosen so as to induce the smallest amount of curvature that can be measured reproducibly to ensure that the curvature relation for plates is accurate. Furthermore, from Eq. 12 and Fig. 1, to minimize the error

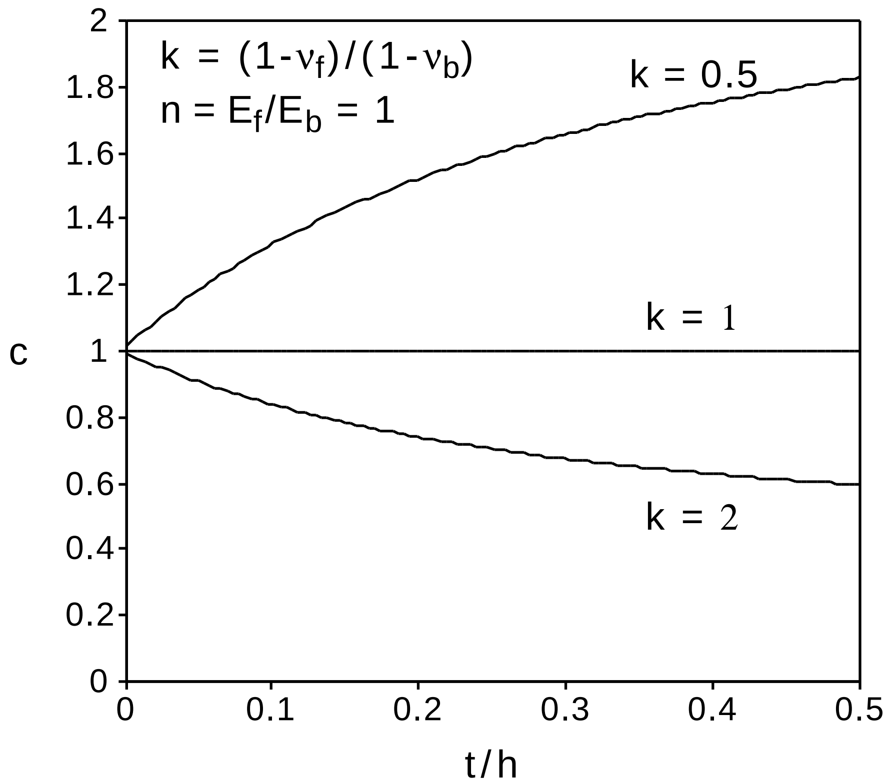

Figure 1. The parameter c from Eq. 12 vs. the film to beam thickness ratio t/h for $n = E_f/E_b = 1$ and three values of $k = (1-\nu_f)/(1-\nu_b)$. When k = 1, c =1 regardless of the value of other parameters.

due to finite thickness of the loading film, it should be chosen such that its Poisson's ratio is as close as possible to that of the cantilever structures.

Single crystal silicon cantilever beams and plates were micromachined from a bonded and etched-back silicon on insulator (SOI) wafer with a 2.5 μm {100}-oriented device layer on top of a 0.5 μm thick thermally grown oxide layer. A 0.1 μm thick layer of silicon nitride was deposited onto the wafer and patterned using standard photolithographic and plasma etching techniques to define the beams and plates. The silicon nitride was then used as a mask for patterning the silicon device layer, using a 44% mixture of potassium hydroxide (KOH) in water. Next, square openings were made in the backside nitride to allow etching of the bulk silicon wafer directly beneath the cantilevers. The KOH etched through the silicon, stopping on the buried oxide film. Finally, the oxide and nitride layers were removed by etching in concentrated hydrofluoric acid, leaving the free-standing 1 mm long x 100 μm wide silicon cantilever beams, and 700 μm x 700 μm plates, as shown in the SEM micrograph in Fig. 2. Each plate was attached to the silicon substrate by a 100

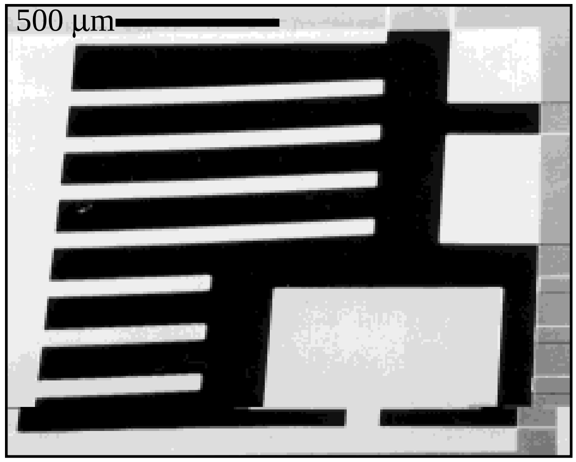

Figure 2. SEM micrograph showing four 1 mm long x 100 μm wide gold-coated silicon cantilever beams, three 500 μm long beams, and a 700 μm x 700 μm square plate attached to the substsrate by a 100 μm x 100 μm square ligament.

μm wide ligament connected at the center of one side. To induce curvature in the beams and plates, the wafer was coated with a 0.035 μm thick evaporated gold film.

A non-contact scanning optical profilometer with 10 nm resolution was used to make the curvature measurements. The UBM Microfocus Measurement System splits an infrared laser beam and focuses the reference beam and reflected beam onto a pair of photodiodes. If the signal from the two beams is unequal, the objective lens height relative to the measurement surface is adjusted with a coil and magnet pair. Accuracy of the instrument was verified with a mechanical stylus profilometer and was found to be better than 1% for a 6 μm step. Because the ratio of curvatures is used to calculate the Poisson's ratio, inaccuracy due to slight miscalibration tends to cancel out. To minimize measurement error, the beams and plates were scanned over the same range of heights, from zero to approximately 20 μm. Considerable error was introduced when, for example, the beams were measured from zero to 20 μm and the plates from zero to -20 μm, or the beams from zero to 35 μm and the plates from zero to 20 μm. Figure 3 shows displacement data from a micromachined cantilever beam and plate. Only data below the cutoff line was used for the

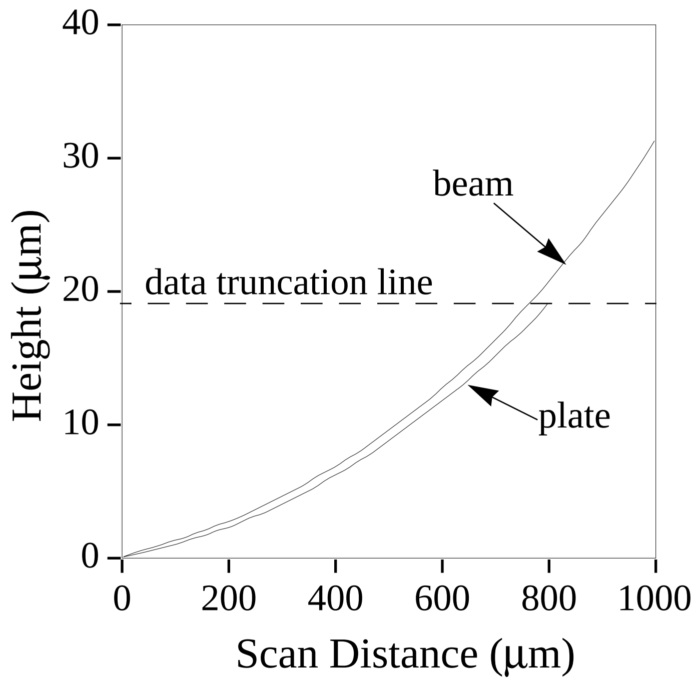

Figure 3. Displacement data from a micromachined cantilever beam and plate. Data within the same height range was used to minimize experimental error.

curvature calculations. Beams were scanned along their lengths in the [110] direction, and the plates were scanned 700 μm along their diagonals. The curvature for the plates should be uniform in all directions because the biaxial modulus is transversely isotropic for {100}-oriented single crystal silicon [12]. Figure 4 shows a two-dimensional scan of a gold-coated plate, confirming the spherical nature of the curvature. A total of seven beams and four plates were scanned, and the least squares method was used to fit the data to a circle.

RESULTS AND DISCUSSION

For the orientation tested ([1 0 0] film normal, [0 1 1] beam axis, [0 $\bar{1}$ 1] contraction direc-

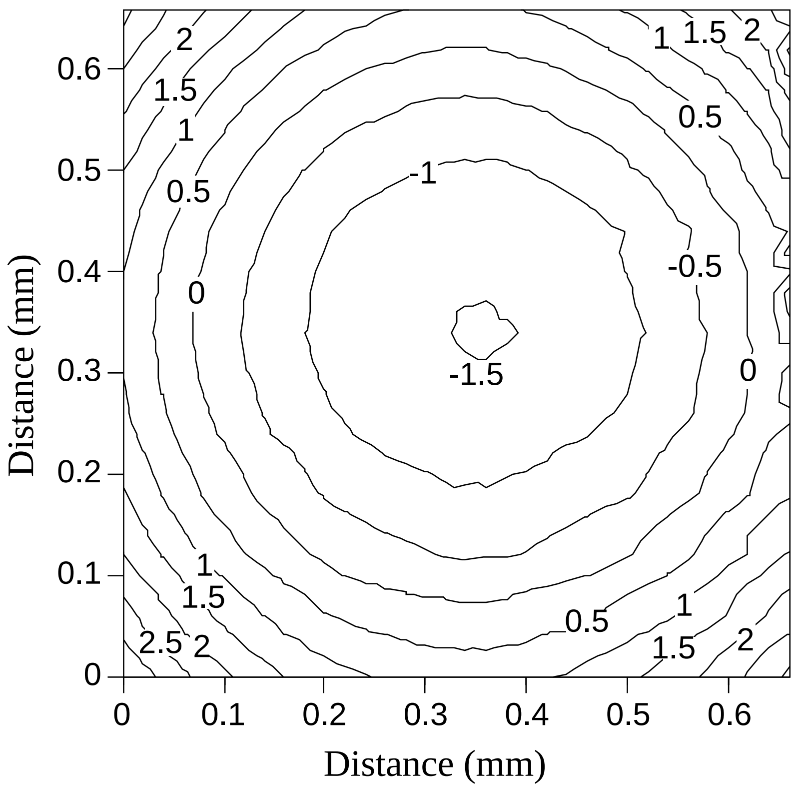

Figure 4. Two-dimensional scan data of a 700 μm square, 2.5 μm thick silicon plate coated with 35 nm gold.

tion) silicon's Poisson's ratio is 0.064 [12]. The measured curvatures and standard deviations were 22.19 ± 0.32 and 23.77 ± 0.69 for the beams and plates, respectively. Using Eq. 8 and taking the standard deviation as the uncertainty, the experimental result for the Poisson's ratio is 0.066 ± 0.043. To validate the use of the thin film curvature relation (Eq. 7), the coefficient c in Eq. 12 is 1.015, using the elastic moduli for bulk gold, E = 70 GPa and ν = 0.42, and single crystal silicon, E = 169 GPa and ν = 0.064, and the corresponding Poisson's Ratio is 0.052.

If careful experimental technique is used and a sufficient number of closely placed beams and plates are measured, it should be possible to measure curvatures with a standard deviation of one or two percent, which, from Eq. 8, results in an uncertainty in the measured Poisson's ratio of ± 0.02 to 0.04. For a typical Poisson's ratio of 0.25, this corresponds to an uncertainty of between 8 and 16 percent.

CONCLUSIONS

Taking the ratio of curvatures of micromachined beams and plates has been shown to be an effective technique for determining the Poisson's ratio of thin films, making it possible to evaluate the second order parameter to within ± 0.02 to 0.04. Using this technique the Poisson's ratio can be determined without knowing the film thickness, residual stress, internal bending moment, or Young's modulus, and depends only on the two measured curvatures. The curvature of the beams and plates can be induced by a gradient in residual stress, or by depositing a thin film under residual stress onto the micromachined structures. For the later case, the thickness of the deposited film must be much less than the beam thickness, or the Poisson's ratios of the two films must be equivalent for the simple curvature relationship to hold.

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